IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Gyanesh P. Khare

Serial No.: 10/021,982 Group Art Unit: 1764

Filed: November 28, 2001 Examiner: James Arnold, Jr.

For: DESULFURIZATION AND NOVEL SORBENT FOR SAME

Rule 131 Declaration

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

1. I, Gyanesh P. Khare, the inventor of the above-referenced patent application, make this declaration to establish actual reduction to practice of the claimed invention in the United States on a date prior to August 25, 1999, which is the effective date of U.S. Patent No. 6,254,766, to Sughrue et al. (hereinafter the "Sughrue '766 Patent") under 35 U.S.C. 102(e). The Examiner in the Office Action dated May 7, 2003 cited the Sughrue'766 patent.

2. Attached hereto is Exhibit A. Exhibit A is a collection of laboratory notebook pages prepared prior to August 25, 1999. Exhibit A evidences acts constituting conception and reduction to practice of a claimed invention. We do not wish to disclose the dates of these materials; therefore, the actual dates have been

blocked out in Exhibit A. The acts evidenced by the materials of Exhibit A were performed in the United States prior to August 25, 1999.

3. The first four pages of the laboratory notebook disclose the method of making of a composition containing silica, alumina, zinc oxide, sodium silicate, and nickel. A mixture comprising alumina, silica, zinc oxide, and sodium silicate was dried, calcined, and then impregnated with nickel nitrate. The composition is then impregnated with a sodium silicate solution. The fifth and sixth pages disclose reducing the composition with hydrogen.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

Gyanesh P. Khare

Gyanesh P. Khare

October 7, 2003

Date

Attachment

EXHIBIT A

[redacted] Repeat of 25161-97 with less H_2O & More Sodium Silicate

Reported material for Spray drying using the following Materials.

P 200.0g Sodium Pyrophosphate
1 1690g DI Water
2 200.0g Vista Dispal 180 - Al_2O_3
3 477g of Celite filter Cell - Silica
788g ZnO
4. 820 Sodium Silicate *
5. Material was no problem to Pump a little on the thick side.
6. Conditions during run were:
7. Atomizer Press 0.3 - 0.38 Bar, operation Shift III
Percent timer 10
8. Inlet Temp 320°c Outlet Temp = 140-145
Air Damper = 5 from Closed. Pump Speed = 3.8
Used the Extend mix nozzel w 0.3 orifice
Material Spray dried Good & Made Very Good material.
9. Will Split Material from bottom Catch (1114g) & dry only 1/2 & Dry & Calcine the other 1/2.
Made 365g in Top Catch.
Des: 38011-6

RECORDED BY B Cass DATE [redacted] WITNESSED BY G.P. Khare DATE [redacted]

[redacted] Sieve Analysis on 38011-6

Rona Sieve analysis on 38011-6 used 50.0g of material that was D+C -50+270.

+50	0g
100	3.14
140	8.87
170	7.03
200	7.82
270	12.04
-270	11.04
	49.94

LBD = 0.74 cc/g
PBD = 0.86 "
PV = 0.4

Note: Material formed a brick after Calcination that broke apart Very Easy.

B. Cass

[redacted]

Impregnated 100.0g of 38011-6 w/ 29.71g of Nickel Nitrate that was dissolved in 22.97g of D.I water. Used a PV of 0.4. This is a 6% Ni loading.

D.S: 38011-6-I-6 LBD = 0.84 PBD = 0.96

See Page 40 for S.S. imp B. Cass

[redacted]

Impregnated 100.0g of 38011-6 w/ 12% Nickel Nitrate. Dissolved 59.42g of Nickel Nitrate in 11.94g of Dist. H2O

D.S: 38011-6-I-12

ATTRITION Test on 5 grams of 38011-6-I-12 sieved on -100/+325 mesh

Filter and wt. 85.44
Stg-1 83.90
1.54 grams

-400 mesh Fines from remaining material in setup 2.39g
2.39
1.54
3.93 ÷ 5 x 100 = 78.6 %

407.24
403.65
3.59

RECORDED BY B. Cass DATE [redacted] WITNESSED BY G. P. Khare DATE [redacted]

[redacted] Impregnation of 38011-6I-6 w Sodium Silicate

Used the Sono Tek atomizer to impregnate 50.0g of 38011-6I-6 that had been heated to 300°F, with a Sodium Silicate Solution made by diluting 20ml of Sodium Silicate with 5ml of DI water.
Sodium Silicate was 9.1% Na_2O & 29.2% SiO_2.
Dried & Calcined in a Stagnt Air oven programed to ramp at 5°C/min. to 120°C, hold 2hrs then ramp to 538°C & hold 1 hr then shut off.

Doc: 38011-40

B. Cass

[redacted] DI Attrition Teston - Y8

Loaded 5.00g of -100 +325 mesh material into Jet Cup.
Reset Air flow to Std rate of 21 L/min. Started heat on tubular.
1.15 Jet Cup Starting wt G = 408.79g filter ending wt G = 84.60
Jet Cup Ending wt G = 407.67 filter Starting wt G = 83.5g
1.12 1.1g
5. 1.12 - 388
Of the 3.88g recovered from the Jet cup 3.20g was +400 mesh.
DI ≃ 36
This material fluidized the same as Three out three runs.

RECORDED BY B. Cass DATE [redacted] WITNESSED BY G. P. Khare DATE [redacted]

Impregnation of 38011-40 with 24% Ni for total of 30% Ni.

[redacted] Donald Engelbert

I impregnated 24 grams of 38011-40 with a solution of 14.26 grams of $Ni(NO_3)_2 \cdot 6H_2O$ and 3.27 grams of dist H_2O.

I put material in oven and ramped the temp 3°C a min to 150°C and held 1 hour. I then ramped the temp ~~to~~ 3°C a min to 635°C and held for 1 hour.

11-3-98 Donald Engelbert

I impregnated above material (26.88 grams) with a solution of 15.97 grams of $Ni(NO_3)_2 \cdot 6H_2O$ and 3 grams of dist H_2O. Dried and Calcined same as above. Des 38011-74.

11-4-98 Donald Engelbert

Davison Index Attrition Test on 38011-74

I loaded 5 grams of 38011-74 sieved on -100/+325 MESH

Filter ending weight = 84.83
" starting weight = 84.19
.64

Jet cup ending weight = 408.30
" " start " = 403.73
4.57

I sieved remaining material in jet cup on 400 mesh sieve
There was .16g ~~remaining~~ DRE. - 400 mesh

.64
.16
.80 ÷ 5 x100 = 16%

DI

RECORDED BY Donald Engelbert DATE [redacted] WITNESSED BY G.P. Kreis DATE [redacted]

[redacted] 25 ml RX Teflon 38011-14

Loaded 10.00g of 38041-74 -100 +325 Mesh into RX & Started heating unit to 700°C under N2.
@ 1120 RX Bed Temp = 709 Started flowing H2

1240 Bed Temp 614 Jacket Temp 598 Start H2 @ 150cc/min and N2 @ 150 cc/min. Start Gasoline.

1340 Bed Temp 639 Jacket Temp 599 Collected 1 hour sample wt = 8.00
Des 38294-58-1-1 Clear
SI = 55

1440 653 599 Collected 2 hour sample wt = 9.63
SI = 70
Des 38294-58-1-2 Clear

1540 669 599 Collected 3 hour sample wt = 9.40
Des 38294-58-1-3 Clear
SI = 60

Shut H2 off Reduced N2 to ~50cc/min Reduced temp to 300

[redacted]

0700 Increased temp setting to 900°. Set N2 flow rate to 240cc/min.

0730 Bed temp = 900, Started Air flowing at 60 cc/min.

0835 Shut Air off & reduced temp setting to 700. Set H2 rate to 300cc/min.

0845 Bed temp = 707 Started H2 at 300 cc/min N2 off

0945 Bed temp = 709 Reduced temp setting to 600°C Reset N2 & H2 flows to 150cc/min

10:30 Bed temp = 621 Jacket = 599 Started H2 & N2 at 150 cc/min each. also Started Gasoline at 13.4 cc/hr.

RECORDED BY B. Cass DATE [redacted] WITNESSED BY G. P. Khare DATE [redacted]

[redacted]

11:30 Bed temp = 647 Jacket = 599 Collected 1 hr Sample wt = 7.80 g
Des: 38294-59-2-1 Clean 5

12:30 Bed temp = 682 " 599 Collected 2 hr Sample wt = 9.35 g Clean
Des: 59-2-2 15

1:30 Bed temp = 679 " 599 Collected 3 hr Sample wt = 9.7 g Clean
Des: 59-2-3 20

2:30 Bed temp = 679 " 599 Collected 4 hr Sample wt = 9.67 Clean
Des: 38294-59-2-4 20

3:30 " = 694 " 599 Collected 5 hr Sample wt = 9.20 Clean
Des: 38294-59-2-5 15

4:00 Shut H2 off + Reduced temp setting to 150°C.

B. Cox

[redacted]

0700 Increased temp setting to 900° Set N2 flow rate to 240cc/min

0730 Bed temp = 900 Started Air flowing at 60cc/min

0830 Shut Air off + reduced temp setting to 700 Set H2 + ~~N2~~ rate to 3800cc/min

0845 Bed temp = 709 Started H2, N2 off

0945 Reduced temp setting to 600° F. Reset H2 + N2 flow rates to 1150cc/min.

10:00 Bed temp = 611 Jacket = 595 Started H2 + N2. Started Gasoline Pump at 13.4cc/hr.

RECORDED BY B. Cox DATE [redacted] WITNESSED BY G. P. Khare DATE [redacted]